

## MEMORANDUM

**TO:** Mr. Addison Rice  
Anderson, Mulholland and Associates

**DATE:** June 29, 2016

**FROM:** R. Infante 

**FILE:** 1605308E

**RE:** Data Validation  
Air samples  
SDG: 1605308E

### SUMMARY

Full validation was performed on the data for several gas samples analyzed for methanol by Compendium Method TO-15. Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters and Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999". The samples were collected at the Bristol Myer Squib facility, Humacao, PR site on May 14, 2016 and submitted to Eurofins Air Toxics, Inc. of Folsom, California that analyzed and reported the results under delivery groups (SDG) 1605308E.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use.

### SAMPLES

The samples included in the review are listed below

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
B7IA-1	1605308E-01A	05/14/2016	Air	Methanol
B7IA-2	1605308E-03A	05/14/2016	Air	Methanol
B7IA-6	1605308E-07A	05/14/2016	Air	Methanol

### REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- o Agreement of analysis conducted with chain of custody (COC) form
- o Holding time and sample preservation
- o Gas chromatography/mass spectrometry (GC/MS) tunes
- o Initial and continuing calibrations
- o Method blanks/trip blanks/field blank

- Canister cleaning certification criteria
- Surrogate spike recovery
- Internal standard performance and retention times
- Field duplicate results
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- Quantitation limits and sample results

## **DISCUSSION**

### **Agreement of Analysis Conducted with COC Request**

Sample reports corresponded to the analytical request designated on the chain-of-custody form except for the following:

- The Chain of Custody (COC) information for sample B7IA-1 did not match the information on the canister with regard to canister identification. The client was notified of the discrepancy and the information on the canister was used to process and report the sample.

### **Holding Times and Sample Preservation**

All samples analyzed within the recommended method holding time. All summa canisters received in good conditions.

### **Initial and Continuing Calibrations**

#### **Methanol by Compendium Method TO-15**

One point calibration performed. Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.

### **Method Blank/Trip Blank/Field Blank**

Target analytes were not detected in laboratory method blanks.

No trip/field blank analyzed with this data package.

### **Laboratory/Field Duplicate Results**

No field/laboratory duplicates were analyzed as part of this data set.

### **LCS/LCSD Results**

#### **Methanol**

No LCS/LCSD (blank spike) was analyzed by the laboratory associated with this data package.

### Quantitation Limits and Sample Results

Dilutions were performed (see worksheet).

Calculations were spot checked.

### Certification

The following samples 1605308E-01A; 1605308E-03A and 1605308E-07A were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document.

  
Rafael Infante  
Chemist License 1888





## Air Toxics

Client Sample ID: B7IA-1

Lab ID#: 1605308E-01A

### EPA METHOD TO-15 GC/MS

File Name:	14052006	Date of Collection:	5/14/16 8:11:00 PM
Dil. Factor:	1.64	Date of Analysis:	5/20/16 11:18 AM

Compound	Rpt. Limit (ppbv)	Amount (ppbv)	Rpt. Limit (ug/m3)	Amount (ug/m3)
Methanol	160	Not Detected	210	Not Detected

Container Type: 6 Liter Summa Canister (100% Certified)

Surrogates	%Recovery	Method Limits
1,2-Dichloroethane-d4	105	70-130
Toluene-d8	102	70-130
4-Bromofluorobenzene	99	70-130



## Air Toxics

Client Sample ID: B7IA-2

Lab ID#: 1605308E-03A

### EPA METHOD TO-15 GC/MS

File Name:	14052007	Date of Collection:	5/14/16 8:25:00 PM
Dil. Factor:	2.07	Date of Analysis:	5/20/16 11:46 AM

Compound	Rpt. Limit (ppbv)	Amount (ppbv)	Rpt. Limit (ug/m3)	Amount (ug/m3)
Methanol	210	Not Detected	270	Not Detected

Container Type: 6 Liter Summa Canister (100% Certified)

Surrogates	%Recovery	Method Limits
1,2-Dichloroethane-d4	103	70-130
Toluene-d8	100	70-130
4-Bromofluorobenzene	99	70-130



## Air Toxics

Client Sample ID: B7IA-6

Lab ID#: 1605308E-07A

### EPA METHOD TO-15 GC/MS

File Name:	14052008	Date of Collection:	5/14/16 8:53:00 PM
Dil. Factor:	2.68	Date of Analysis:	5/20/16 12:19 PM

Compound	Rpt. Limit (ppbv)	Amount (ppbv)	Rpt. Limit (ug/m3)	Amount (ug/m3)
Methanol	270	Not Detected	350	Not Detected

Container Type: 6 Liter Summa Canister (100% Certified)

Surrogates	%Recovery	Method Limits
1,2-Dichloroethane-d4	104	70-130
Toluene-d8	101	70-130
4-Bromofluorobenzene	99	70-130



Relinquishing signature on this document indicates that sample is being shipped in compliance with all applicable local, State, Federal, national, and international laws, regulations and ordinances of any kind. Air Toxics Limited assumes no liability with respect to the collection, handling or shipping of these samples. Relinquishing signature also indicates agreement to hold harmless, defend, and indemnify Air Toxics Limited against any claim, demand, or action, of any kind, related to the collection, handling, or shipping of samples. D.O.T. Hotline (800) 467-4922

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<b>Project Info:</b>  P.O. # _____  Project # _____  Project Name _____	<b>Turn Around Time:</b>  <input type="checkbox"/> Normal <input checked="" type="checkbox"/> Rush <b>1-Day</b> <i>specify</i>	<i>Lab Use Only</i>  <b>Pressurized by:</b>  Date: _____  Pressurization Gas: _____ <div style="text-align: right;">N<sub>2</sub>    He</div>
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Lab I.D.	Field Sample I.D. (Location)	Can #	Date of Collection	Time of Collection	Analyses Requested	Canister Pressure/Vacuum			
						Initial	Final	Receipt	Final (psi)
01a	B7 IA-1	00300	05/14/16	2011	T0-15	+30	7.5		
02a	B7 IA-1D	00355	05/14/16	2012	T0-15	+30	7.0		
03a	B7 IA-2	N0593	05/14/16	2025	T0-15	+30	13.0		
04a	B7 IA-3	00314	05/14/16	2034	T0-15	+30	10.0		
05a	B7 IA-4	5737	05/14/16	2057	T0-15	+30	6.5		
06a	B7 IA-5	6L1224	05/14/16	2004	T0-15	+30	6.5		
07a	B7 IA-6	00316	05/14/16	2053	T0-15	+30	~17		

Relinquished by: (signature) Date/Time

Received by: (signature) Date/Time

Notes:

Relinquished by: (signature) Date/Time

Received by: (signature) Date/Time

Relinquished by: (signature) Date/Time

Received by: (signature) Date/Time

Lab Use Only

Shipper Name

Air Bill #

Temp (°C)

Condition

Custody Seals Intact?

Work Order #

received samples 01a, 03a, 07a 5/18/16

0059 of 0070

# DATA REVIEW WORKSHEETS

Project Number: 1605308E  
Date: 05/14/2016

## REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: QC criteria from "Compendium Method TO-15. Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters and Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999"; USEPA Hazardous Waste Support Branch. Validating Air Samples. Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006). The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Eurofins - Air Toxics data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: 1605308E Sample matrix: Air  
No. of Samples: 3

Trip blank No.: -  
Field blank No.: -  
Equipment blank No.: -  
Field duplicate No.: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input checked="" type="checkbox"/> GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input checked="" type="checkbox"/> Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input type="checkbox"/> N/A Matrix Spike/Matrix Spike Duplicate	

Overall Comments: Methanol by method TO-15

### Definition of Qualifiers:

J- Estimated results  
U- Compound not detected  
R- Rejected data  
UJ- Estimated nondetect

Reviewer: Rafal Lukut  
Date: 06/29/2016



## DATA REVIEW WORKSHEETS

## DATA COMPLETENESS

### MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below       

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time. All summa canisters received in good conditions. The Chain of Custody (COC) information for sample B7IA-1 did not match the information on the canister with regard to canister identification. The client was notified of the discrepancy and the information on the canister was used to process and report the sample.				

### Criteria

Aqueous samples – 14 days from sample collection for preserved samples (pH ≤ 2, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C, no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: 4 ± 2 °C): N/A – summa canisters

### Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ).

If the % solid of soil samples is < 10%, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted (> 10°C), estimate positive results (J) and nondetects (UJ).

All criteria were met   X    
 Criteria were not met see below

## GC/MS TUNING

X The BFB performance results were reviewed and found to be within the specified criteria.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

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## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 05/20/2016  
 Dates of continuing calibration: 05/20/2016  
 Instrument ID numbers: MSD-14  
 Matrix/Level: Air/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
One point calibration. Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.					

#### Criteria

All RFs must be  $> 0.05$  regardless of method requirements for SPCC.  
 All %RSD must be  $\leq 15\%$  regardless of method requirements for CCC.  
 All %Ds must be  $\leq 30\%$  regardless of method requirements for CCC.  
 Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of  $\geq 0.995$  has therefore been utilized as professional judgment.

#### Actions

If any compound has an initial RF or a continuing RF of  $< 0.05$ , estimate positive results (J) and reject nondetects (R), regardless of method requirements.  
 If any compound has a %RSD  $> 15\%$ , estimate positive results (J) and use professional judgment to qualify nondetects.  
 If any compound has a %RSD  $> 90\%$ , estimate positive results (J) and reject nondetects (R).  
 If any compound has a % D  $> 30\%$ , estimate positive results (J) and reject nondetects (R).  
 If any compound has a % D  $> 30\%$ , estimate positive results (J) and nondetects (UJ).  
 If any compound has a % D  $> 90\%$ , estimate positive results (J) and reject nondetects (R).  
 If any compound has  $r < 0.995$ , estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

**V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)**

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

### Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/MATRIX	COMPOUND	CONCENTRATION UNITS
		All_method_blank_meeth_method_specific_criteria		

Field/Equipment/Trip blank

[illegible]

All criteria were met X  
Criteria were not met  
and/or see below

## Blank Actions

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

**Specific actions are as follows:**

If the concentration is  $\geq$  SQL but  $\leq$  AL, report the compound as not detected (U) at the reported concentration.

If the concentration is  $\geq$  SQL and  $>$  AL, report the concentration unqualified.

**Notes:**

**High and low level blanks must be treated separately**

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

[illegible]

# DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

## SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND			ACTION
	1,2-DICHLOROETHANE-d4	Toluene-d8	4-BFB	

  Surrogate recoveries within laboratory control limits  

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

QC Limits\* (Air)

      LL\_to\_UL      70\_to\_130                  70\_to\_130      70\_to\_130      

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below N/A

### VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

#### 1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ Matrix/Level: \_\_\_\_\_

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
____MS/MSD_are_not_required_as_part_of_Method_TO-15;_blank_spike_used_to_assess____					
____accuracy____					

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (JJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.



## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
Criteria were not met \_\_\_\_\_  
and/or see below N/A

## VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

### MS/MSD – Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ Matrix/Level/Unit: \_\_\_\_\_

COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION
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**Actions:**

- \* If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).  
\* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?  
 Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
No LCS/LCSD (Blank spike) analyzed in this data package.			

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

#### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

## DATA REVIEW WORKSHEETS

All criteria were met   N/A    
 Criteria were not met  
 and/or see below           

### IX. LABORATORY DUPLICATE PRECISION

Sample IDs:                    -                   

Matrix:        -       

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD  $\pm$  25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
No field/laboratory duplicate analyzed with this data package.					

#### Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

# DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below       

## X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- \* Area of +40% or -40% of the IS area in the associated calibration standard.
- \* Retention time (RT) within  $\pm 0.06$  seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
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Internal standard area and retention times within laboratory control limits for both samples and calibration standards


Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%		IS AREA > + 40%
Positive results	J		J
Nondetected results	R		ACCEPT

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met  
and/or see below       

### XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

1605308E-03A

4-Bromofluorobenzene      RF = 0.52579

$$[ ] = (324997)(400)/(626756)(0.52579)$$

$$= 394.5 \text{ ppbv OK}$$

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

**A. Dilution performed**

[illegible]

List samples which have  $\leq 50\%$  solids

[illegible]

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)